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SUPPLEMENT TO EFFECTS OF POLLUTION ON WATER QUALITY, PERDIDO RIVER AND BAY, ALABAMA AND FLORIDA

Environmental Protection Agency
Athens, Georgia

February 1971

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SUPPLEMENT TO

EFFECTS OF POLLUTION ON WATER QUALITY

PERDIDO RIVER AND BAY

ALABAMA AND FLORIDA

Environmental Protection Agency
Water Quality Office
Southeast Water Laboratory
Technical Services Program
Athens, Georgia

February 1971

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INTRODUCTION

BACKGROUND

On April 7, 1969, the Director, Technical Staff, Alabama Water Improvement Commission, requested technical assistance from the Southeast Region of the Federal Water Pollution Control Administration in evaluating water quality conditions and waste sources in Perdido Bay. The bay and its tributaries were studied during September 1969, and an evaluation of the data showing interstate pollution (primarily from the St. Regis paper mill at Cantonment, Florida) was presented in a report entitled Effects of Pollution on Water Quality, Perdido River and Bay, Alabama and Florida. In January 1970, the report was presented at an enforcement conference, called at the request of Governor Albert P. Brewer of Alabama, to consider the matter of pollution of interstate waters of Perdido Bay and its tributaries (Florida-Alabama). With minor changes, the recommendations outlined in the 1970 report were adopted by the Conferees. On the basis of more thorough studies conducted at the mill in November 1970, revisions in the effluent recommendations relating to waste discharges from the St. Regis Mill are now suggested.

PURPOSE AND OBJECTIVES

Following the 1970 enforcement conference, it was determined that another survey of waste discharges at the St. Regis Mill was needed to supplement data obtained in the 1969 field study. The reasons for the survey were: (a) to confirm previous results or produce more representative

values of average mill discharges, and (b) to evaluate the strength of certain process wastes generated within the mill.

ACKNOWLEDGEMENTS

The cooperation and contribution of the Florida Department of Air & Water Pollution Control and the St. Regis Paper Company are gratefully appreciated. For their assistance, we extend special thanks to:

- The Florida Department of Air & Water Pollution Control's Regional Office for providing laboratory space and for the suspended solids analyses.

- The St. Regis Technical Department for assisting in sampling point selection and sample collection.

SUMMARY

During November 1970, personnel from the Southeast Region, Federal Water Quality Administration (FWQA) conducted a 72-hour survey of waste discharges at the St. Regis Integrated Pulp and Paper Mill at Cantonment, Florida. The mill produces 950 tons per day of pulp, of which approximately 280 tons per day are bleached.

At the time of the survey, two settling basins normally used in the waste treatment process were not in use. Settled solids were in the process of being removed from the basins and wastes were routed through a temporary settling basin. Treatment facilities at the time of the survey, then, included a settling basin with approximately 18 hours detention time followed by two lakes with a combined estimated detention time of approximately 60 hours. Riffle terraces are located before and after the final lake, and foam barriers are placed across the channel below the riffle terraces. The temporary changes in the treatment process had no effect on the raw waste load. Approximately 29 million gallons per day (mgd) of liquid wastes are discharged into Elevenmile Creek. Survey results showed that:

1. Wastes containing approximately 73,600 pounds per day of 5-day biochemical oxygen demand (BOD_5) are generated within the mill. The temporary treatment system removed 42% of the average 5-day biochemical oxygen demand load, leaving 58% or 42,500 pounds per day in the discharge to Elevenmile Creek. The discharged effluent is equivalent to a population of 250,000.

2. The total organic carbon (TOC) load before treatment is calculated to be 68,600 pounds per day. Treatment in effect at the time removed 29% of the TOC load, leaving 48,800 pounds of total organic carbon in the discharge to the receiving stream.

3. Approximately 59,400 pounds per day of suspended solids enter the St. Regis treatment facilities. Treatment removed 71% of the suspended solids, leaving 17,100 pounds per day in the final effluent.

4. Foam collected from the Alabama shore of Perdido Bay contained the same chemical constituents found in foam collected from the St. Regis treated effluent at Florida Highway S-186.

CONCLUSIONS AND RECOMMENDATIONS

CONCLUSIONS

1. Data collected during the 72-hour waste survey agree with long term average values recorded by the St. Regis Paper Company.

2. The St. Regis Paper Company's Mill located north of Pensacola, at Cantonment, Florida is generating an excessive amount of raw pulp and paper mill wastes. Approximately 29 mgd of inadequately treated wastes are discharged into Perdido Bay via Elevenmile Creek.

   Information compiled from a waste survey of mills in the Southern Pulp and Paper Industry indicated that for the amount of bleached and unbleached pulp and paper produced at the St. Regis Mill, the average amount of wastewater volume and BOD_5 loading should be approximately 23.0 mgd and 50,900 pounds per day respectively. The wastewater volume and BOD_5 loads actually generated exceed the average southern Kraft operations values by 20% and 31% respectively. In-plant process improvement is clearly indicated.

3. The St. Regis paper mill waste effluent is the major cause of the foam problem in Perdido Bay.

RECOMMENDATIONS

The recommendations of the Conferees at the Federal-State enforcement conference on pollution of Perdido River and Bay, held in January 1970 were as follows:

To abate the existing pollution in Perdido River and Bay, the following water quality management and waste abatement program is recommended as a minimum:

1. There shall be an overall removal efficiency of 90% for carbonaceous waste material from the St. Regis Paper Company. The St. Regis waste effluent, as measured below the present riffle terraces, shall not exceed 8,800 pounds per day of five-day BOD, and 2,610 pounds per day of total organic carbon. All settleable solids shall be removed.

2. The St. Regis Paper Company shall remove the foam causing constituents from its effluent.

3. The St. Regis Paper Company shall reduce the color of its waste discharge to meet the water quality standards of Florida, Alabama and the Federal Government.

4. St. Regis Paper Company shall make a feasibility study of construction of an essentially closed system involving recirculation, treatment and reuse of its process water. A progress report shall be submitted to the Conferees by January 1, 1971.

5. Sewage treatment plants on Bayou Marcus shall be consolidated into a central facility with removal efficiencies of 90% for carbonaceous material plus adequate disinfection of effluent on a year round basis.

6. All waste abatement facilities shall be in operation not later than December 31, 1972.

On the basis of information now available, it is recommended that the Conferees consider the following changes in Item 1 of their original recommendations:

1a. There shall be an overall removal efficiency of 90% for carbonaceous waste material from the St. Regis Paper Company. All settleable solids shall be removed.

b. In view of the excessive amount of raw waste load generated at the plant, the St. Regis Paper Company shall make a feasibility study and submit plans for in-plant process changes or other measures to meet an industry-wide average effluent discharge value, which in this case would result in an allowable waste load discharge of 5,100 pounds per day of BOD_5 following 90% treatment. The allowable effluent TOC load shall correspond to 90% TOC removal following in-plant load reduction and in no case shall be greater than 6,860 pounds per day.

DESCRIPTION OF STUDY AND STUDY AREA

STUDY AREA

The St. Regis Paper Company operates an integrated pulp and paper mill in Cantonment, Escambia County, Florida. The mill is located approximately 12 miles north of Pensacola and has been in operation since 1941. The original mill has been expanded to essentially two mills at one site which presently produce approximately 950 tons per 24-hours of bleached and unbleached pulp by the sulfate (Kraft) process. Approximately 280 tons per day of pulp are bleached. Pulp is converted into approximately 885 tons per day of bleached and unbleached products. The 29 mgd of liquid wastes generated at the plant are discharged into Perdido Bay via Elevenmile Creek.

Treatment facilities at the mill normally consist of two settling basins followed by two small lakes. However, at the time of the survey, treatment facilities included only a temporary settling basin with an estimated 18 hours detention time, followed by the two lakes with an additional combined detention time of approximately 60 hours (Figure 1). The two out-of-service settling basins normally provide a detention time of 32 hours. Riffle terraces are located before and after the final lake, and foam barriers are placed across the channel below the riffle terraces.

STUDY PROGRAM

To supplement grab sample data obtained from the September 1969 survey, a 72-hour survey of sources of wastes and waste treatment efficiency at the mill was conducted during the period November 10-13, 1970. A total of

10 sites were selected for sample collection. The 10 sampling points included most major waste sources (see Table 1).

Since the original mill and expansions have resulted in a sewer system which serves essentially two mills at one site, in-plant waste sources are numerous, and it is difficult to represent each source with a limited number of sampling stations. Combined raw waste effluent from all processes is easily measurable at one point, however. Figure 2 is a waste flow diagram of the mill which shows the numerous waste sources and sampling stations routinely sampled by mill management. With the assistance of the mill's engineering technical division, eight in-plant sampling points were selected to measure the major waste sources. Two other points were selected (R-1 & R-2) to represent combined plant waste before and after treatment. Figure 3 shows waste sources and related stations selected for sample collection.

Analyses made at the plant included BOD_5, TOC and suspended solids. Since the Conferees' recommendations called for the removal of all settleable solids, suspended solids concentrations were determined to indicate the present degree of solids removal.

Acid characterization of foam found in Perdido Bay was compared with that found in the mill effluent.

WASTE TREATMENT FACILITIES
ST. REGIS PAPER COMPANY – CANTONMENT, FLORIDA
NOVEMBER 10 – 13, 1970

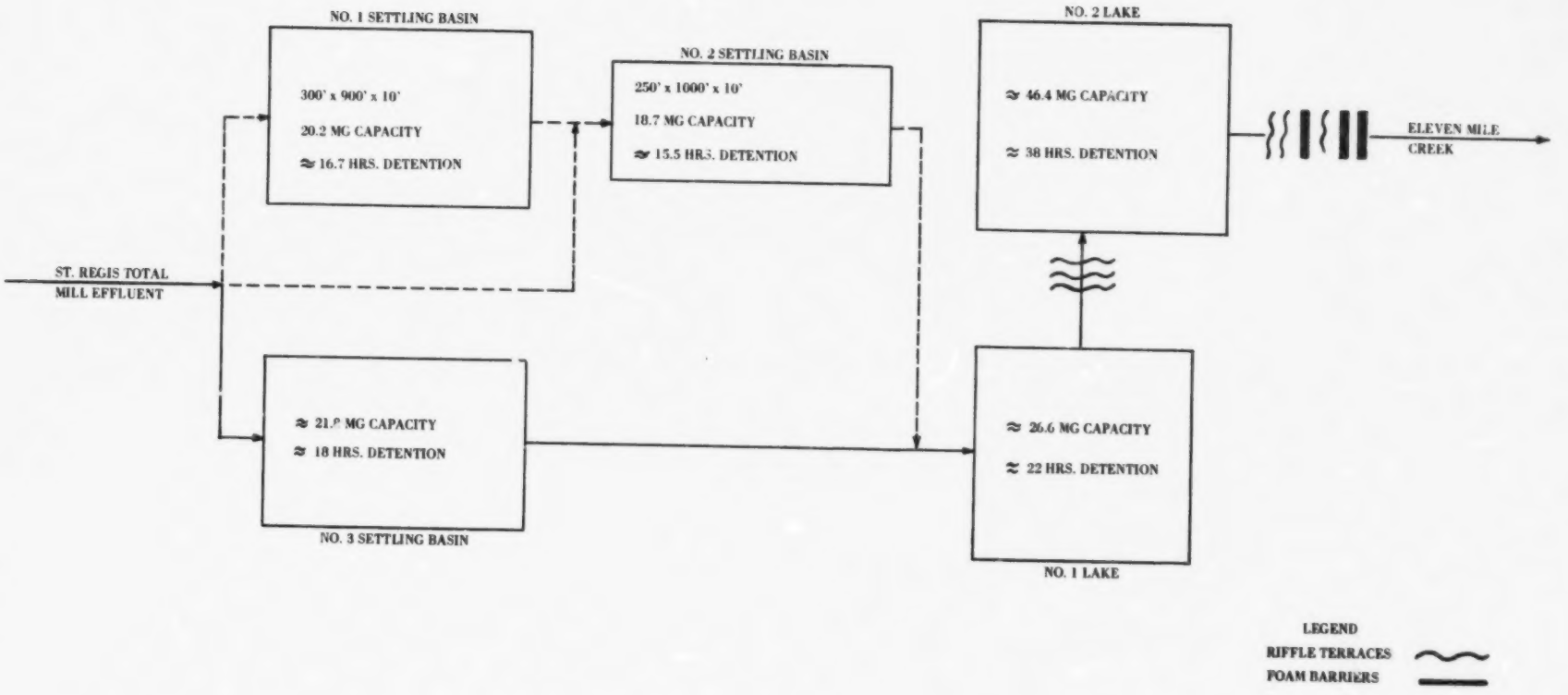
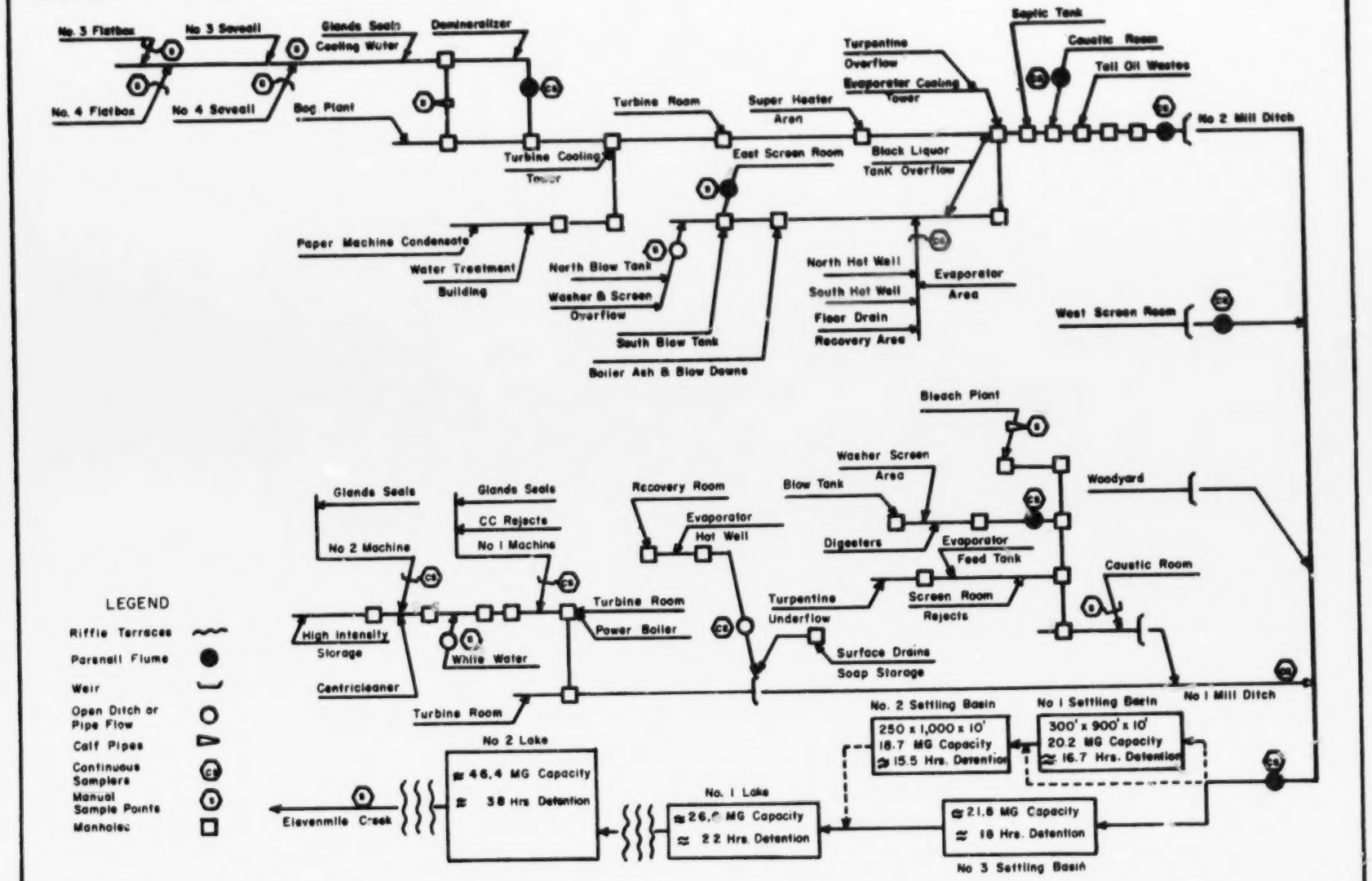


Fig. 2

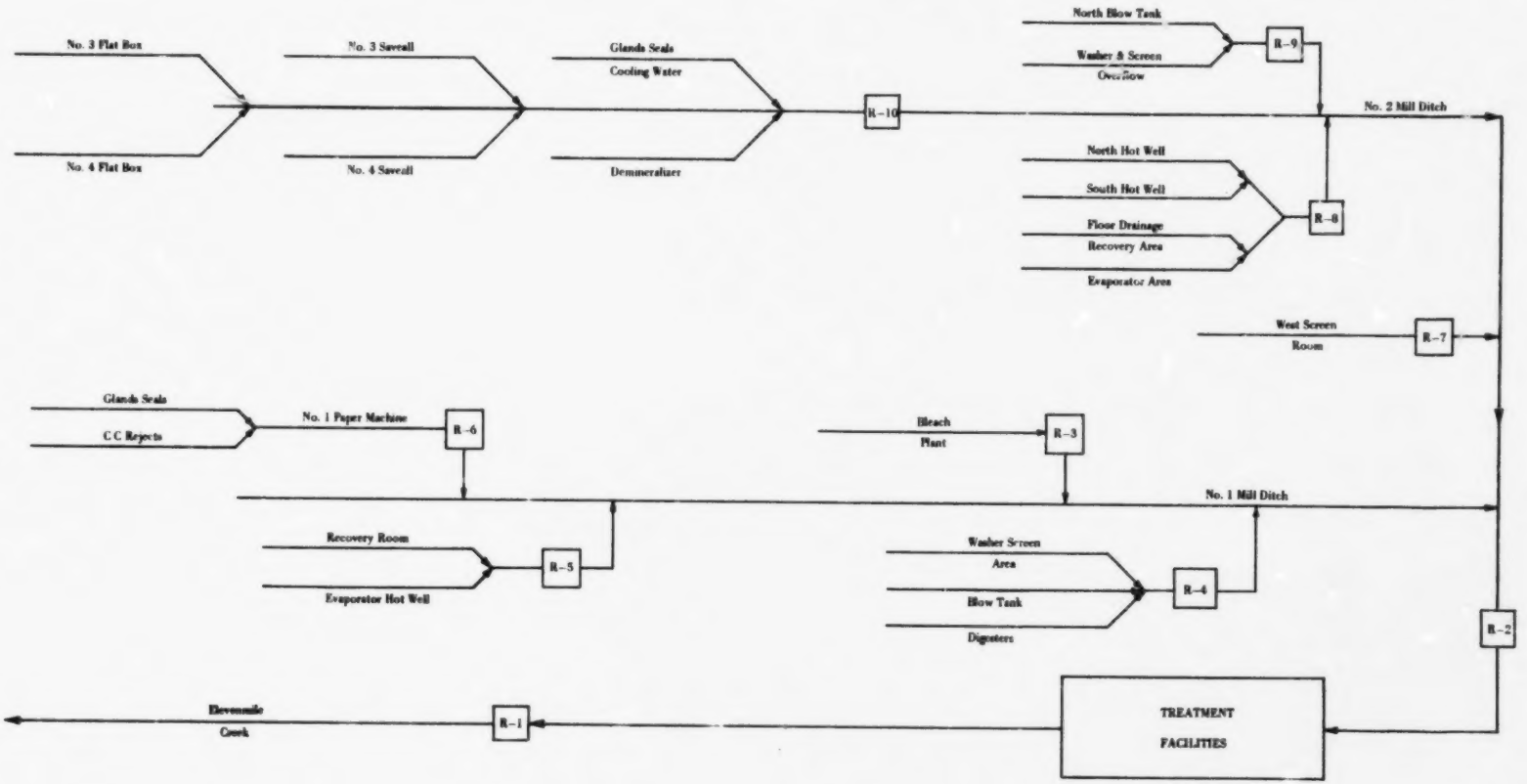
WASTE FLOW DIAGRAM
ST. REGIS PAPER CO. - CANTONMENT, FLA.



B-6

Fig. 3

WASTE FLOW SAMPLING DIAGRAM
ST. REGIS PAPER COMPANY, CANTONMENT, FLA.
NOVEMBER 10 – 13, 1970



9-C

TABLE 1

SAMPLING STATION DESCRIPTIONS

Station Number	Location
R-1	Treated effluent. Elevenmile Creek at Florida Highway S-186 located in NE 1/4 sec. 3, T. 1S, R. 31W, Escambia County, Florida.
R-2	Combined plant raw waste effluent.
R-3	Bleach plant effluent.
R-4	No. 1 mill washer screen area, blow tank and digester effluent.
R-5	No. 1 mill evaporator and recovery room effluent.
R-6	No. 1 paper machine effluent.
R-7	West screen room effluent.
R-8	No. 2 mill evaporator and recovery area effluent.
R-9	No. 2 mill north blow tank and washer and screen overflow effluent.
R-10	No. 2 mill effluent at paper machine Nos. 3 and 4.

STUDY FINDINGS

The chemical data at all in-plant sampling stations compare favorably with values normally observed by St. Regis personnel. Table 2 shows a summary of weighted average concentrations and calculated loads for each of the three parameters measured (BOD_5, TOC & Suspended Solids). The combined plant effluent data (before and after treatment) are representative of long term averages recorded by the mill.

Table 3 shows in-plant station waste loadings and combined plant loadings before and after treatment. Although each in-plant source was not measured, sampling did give an accurate account of treatment efficiencies and total waste loads generated within the mill and discharged into Elevenmile Creek.

Data obtained from the survey were compared with wastewater volumes and waste loads that may be expected from average southern Kraft operations. The Water Resources Research Institute at the University of North Carolina recently issued a publication which included results of a comprehensive survey of the amount of waste and wastewater generated per ton of production for different types of mills in the southern pulp and paper industry.[1/] This publication shows that the average volume of wastewater per ton of product is 18,500 gallons for unbleached product and 38,000 gallons for bleached product. The St. Regis Mill produces approximately 670 tons per day of unbleached pulp and 280 tons per day of bleached pulp. Reported wastewater flow during the survey was about 29 mgd. This exceeds the average amount for southern Kraft operations by approximately 6 mgd.

1/ Survey of Water Utilization and Waste Control Practices in the Southern Pulp and Paper Industry, Water Resources Research Institute, University of North Carolina, June 1970.

TABLE 2

CHEMICAL DATA SUMMARY

ST. REGIS PAPER COMPANY - CANTONMENT, FLORIDA

STUDY - NOVEMBER 10-13, 1970

Station No.	Flow	Five-Day Biochemical Oxygen Demand		Suspended Solids		Total Organic Carbon	
		(mg/l)*	(lbs/DAY)	(mg/l)*	(lbs/DAY)	(mg/l)*	(lbs/DAY)
R-1	28.94	176	42,500	71	17,100	202	48,800
R-2	28.94	305	73,600	246	59,400	284	68,600
R-3	5.32	124	5,500	125	5,600	203	9,000
R-4	0.33	545	1,500	99	272	415	1,100
R-5	2.87	247	5,900	16	383	173	4,100
R-6	2.13	104	1,800	168	3,000	106	1,900
R-7	0.30	548	1,400	190	475	374	936
R-8	1.19	705	6,700	157	1,600	598	5,900
R-9	0.42	797	2,800	238	834	790	2,800
R-10	4.71	80	3,100	80	3,100	67	2,600

NOTE: * Flow weighted averages.

TABLE 3

DISTRIBUTION OF WASTE LOADS SAMPLED

ST. REGIS PAPER COMPANY - CANTONMENT, FLORIDA

Station No.	Flow		BOD_5 Load		TOC Load		Suspended Solids Load	
	(mgd)	% Total	#/day	% Total	#/Day	% Total	#/Day	% Total
R-1	28.94	100	42,500	58	48,800	71.1	17,100	28.9
R-2	28.94	100	73,600	100	68,600	100.0	59,400	100.0
R-3	5.32	18.4	5,500	7.5	9,000	13.1	5,600	9.3
R-4	0.33	1.1	1,500	2.0	1,100	1.7	272	0.5
R-5	2.87	9.9	5,900	8.0	4,100	6.0	384	0.6
R-6	3.13	10.8	1,900	2.5	1,900	2.7	3,000	5.0
R-7	0.30	1.0	1,400	1.9	936	1.4	475	0.8
R-8	1.19	4.1	6,700	9.1	5,900	8.7	1,600	2.6
R-9	0.42	1.4	2,800	3.8	2,800	4.0	834	1.4
R-10	4.71	16.3	3,100	4.3	2,600	3.8	3,100	5.3
In-Plant Coverage	18.27	63.1	28,800	39.1	28,400	41.4	15,200	25.5

BIOCHEMICAL OXYGEN DEMAND

Although in-plant sampling accounted for only 39% of the BOD_5 load entering the treatment basins, approximately 63% of the total flow was accounted for. A total account of waste load from each plant process was not intended in the time available. The BOD_5 concentrations during the November 10-13 study period are shown in Table 4.

The average BOD results at sampling stations R-1 and R-2 (before and after treatment sampling stations) showed very little variation among individual samples and are in close agreement with eight-month averages reported by mill management.

The average raw waste load generated within the plant is calculated to be 73,600 pounds per day of BOD_5. Treatment in effect reduced this load to 42,500 pounds per day, a removal of 42%. The untreated waste load of 73,600 pounds per day is 15,200 pounds per day less than the 5-day BOD measured during the more limited September 1969 survey. Treatment efficiency, however, did not vary a great deal from the 38% reported in the earlier survey.

The Water Resources Research Institute publication reports average BOD_5 loads for unbleached and bleached pulp and paper mills to be 43 and 79 pounds per ton of production respectively. Using the average values, a raw load of 50,900 pounds per day of BOD_5 was calculated. The present raw load generated exceeds the calculated load by 22,700 pounds. This excessive raw waste load at St. Regis indicates that in-plant process improvements should be effected.

TABLE 4

FIVE-DAY BIOCHEMICAL OXYGEN DEMAND DATA (mg/l)

ST. REGIS PAPER COMPANY - CANTONMENT, FLORIDA

NOVEMBER 10-13, 1970

Date (1970) Nov.	Work Shift Hours	STATION NUMBERS R-1	R-2	R-3	R-4	R-5	R-6	R-7	R-8	R-9	R-10
10	7-3	141	272	115	590	375	-	700	1040	1040	-
10	3-11	166	260	126	360	345	-	625	640	605	-
11	11-7	169	272	75	365	222	81	520	610	860	89
11	7-3	172	268	122	360	145	-	295	415	745	-
11	3-11	184	415	115	690	288	-	470	680	750	-
12	11-7	158	365	104	640	242	126	725	710	650	70
12	7-3	220	258	93	510	240	-	540	760	1380	-
12	3-11	210	348	230	280	196	-	520	1000	590	-
13	11-7	170	292	127	375	174	-	405	485	580	-
	MEAN	177	306	123	463	247	104	533	704	800	80
WT. AVERAGE*		176	305	124	545	247	104	548	705	797	80

NOTE: * Flow Weighted Average

TOTAL ORGANIC CARBON

The total organic carbon (TOC) test is a useful means of measuring the loading and treatment efficiency of organic waste producing plants such as pulp and paper mills. The TOC test together with the BOD test provides an excellent measure of industrial waste pollution. However, only limited TOC data for paper mill operations are presently available, and average values that may be expected from pulp and paper mill processes have not been developed.

Since a specific TOC recommendation was made by the Conferees, additional TOC data before and after treatment were deemed necessary to provide the representative raw loading values needed before a 90% reduction value can be recommended.

The TOC loadings at all sampling stations closely paralleled BOD_5 results. Average TOC loadings at critical stations R-1 and R-2 are shown in Table 2. The summarized TOC data presented in Table 2 were calculated from the weighted average concentrations shown in Table 5. The summarized data show that 68,600 pounds per day of TOC are generated in the mill, with an effluent load after treatment of 48,800 pounds per day.

The Conferees recommended that the TOC discharge to Elevenmile Creek be restricted to 2,610 pounds per day. The more comprehensive 1970 survey, however, showed that 6,860 pounds per day of TOC in the final effluent is more representative of the present waste load following 90% TOC reduction. In the preceding BOD section, in-plant changes to reduce the untreated BOD_5 load to a more acceptable level is discussed. In-plant improvements will also reduce the untreated TOC load. No industry-wide averages are available at this time to indicate what this acceptable TOC effluent level should be, but it would be less than 6,860 per day.

TABLE 5

TOTAL ORGANIC CARBON DATA (mg/l)

ST. REGIS PAPER COMPANY - CANTONMENT, FLORIDA

NOVEMBER 10-13, 1970

Date (1970)	Work Shift Hrs	STATION NUMBERS R-1	R-2	R-3	R-4	R-5	R-6	R-7	R-8	R-9	R-10
Nov.											
10	7-3	170	310	180	450	215	35	330	990	1250	80
10	3-11	200	230	180	160	330	40	440	425	475	50
11	11-7	200	240	125	300	125	50	395	615	525	75
11	7-3	205	225	140	305	130	260	350	245	650	80
11	3-11	170	350	205	540	165	45	320	540	670	80
12	11-7	195	365	265	500	146	285	450	650	740	80
12	7-3	215	260	260	395	150	70	395	670	1550	65
12	3-11	235	375	355	145	130	40	350	800	535	45
13	11-7	225	205	145	350	165	30	245	445	740	45
	MEAN	202	284	206	349	173	95	364	598	793	67
	WT. AVERAGE*	202	284	203	415	173	106	374	598	790	67

*Flow weighted average.

TOTAL SUSPENDED SOLIDS

The Conferees recommended that all settleable solids be removed from the discharged effluent. Since samples collected during the initial survey were not analyzed for solids, it was desirable to gather information on the capability of the treatment facilities to remove settleable materials. Arrangements were made with the Florida Department of Air and Water Pollution Control to make total suspended solids determinations.[2/] Results of these determinations are summarized in Table 6.

Survey data show that approximately 59,400 pounds per day of total suspended solids enter the St. Regis treatment facilities. Treatment removed 71% of the total suspended solids, leaving 17,100 pounds per day in the final effluent. Although the suspended solids removal of 71% is higher than expected for the lagoon system, removal would approach 90% in a mechanically cleaned clarifier. Accumulated solids present a removal problem in settling lagoons and will interfere with the effectiveness of such lagoons as secondary treatment devices. A mechanically cleaned primary basin preceding biological treatment is normally preferred.

FOAM

Resin acids, together with fatty acids, form the principal constituents of the tall oil produced as a by-product of the sulfate pulping process. Pimaric, isopimaric and dehydroabetic acid are the types of resin acids found in paper mill wastes. Myristic, palmitic, oleic and linoleic

[2/] This type analysis is not equivalent to settleable solids determination but provides insight into solids removal.

TABLE 6

SUSPENDED SOLIDS DATA (mg/l)

ST. REGIS PAPER COMPANY - CANTONMENT, FLORIDA

NOVEMBER 10-13, 1970

Date (1970)	STATION NUMBERS									
	R-1	R-2	R-3	R-4	R-5	R-6	R-7	R-8	R-9	R-10
Nov.										
10-11	50	272	54	134	23	38	109	261	54	24
11-12	78	298	44	108	14	320	384	54	414	126
12-13	84	170	272	70	10	122	140	156	244	90
MEAN	71	247	123	104	16	160	211	157	237	80
WT.AVG.*	71	246	125	99	16	168	190	157	238	80

*Flow weighted averages.

acid are typical of the types of fatty acids normally observed in wastes from the tall oil process. Foam samples collected from mill effluent and Perdido Bay during the initial survey were determined to be of paper mill origin. To supplement the data obtained from limited sampling during the earlier survey, foam samples were again collected from mill effluent and Perdido Bay and analyzed for their resin and fatty acids content. Table 7 lists results of the acid analyses.

Foam samples were collected from two locations within the mill and from the treated effluent during the survey. Only light isolated traces of foam were observed in Elevenmile Creek downstream from Florida Highway S-186 and on Perdido Bay during the three-day study. On December 8, 1970, residents along the bay complained of excessive foaming, and samples of the foam were collected at Lillian Bridge and Manuel Point at that time. Chemical constituents of foam collected from the Alabama shore of Perdido Bay contained chemical constituents identical to foam collected from the St. Regis treated effluent at Florida Highway S-186. The similarity of the acids content in the foam samples indicate that the St. Regis effluent is the major cause of the foam problems observed in Perdido Bay.

TABLE 7

FOAM ANALYSIS
ST. REGIS EFFLUENT AND PERDIDO BAY

Station No. and Location	Date (1970)	FATTY ACIDS Myristic (mg/l)	Palmitic (mg/l)	Oleic (mg/l)	Linoleic (mg/l)
R-1, Elevenmile Creek at Fla. Hwy. S-186	11/10	*	0.16	0.57	0.38
R-1, " "	11/12	*	0.96	4.7	4.0
No. 2 Mill Recovery Area Effluent	11/10	3.0	9.6	123	47
" " " "	11/12	*	41	280	150
No. 1 Total Mill Ditch	11/12	*	17	*	*
Perdido Bay at Lillian Bridge	12/8	0.069	0.11	ND	ND
Perdido Bay ½ mi. Below Manuel Point	12/8	ND	0.10	ND	ND

Station No. and Location	Date (1970)	RESIN ACIDS Pimaric (mg/l)	Isopimaric (mg/l)	Dehydroabetic (mg/l)
R-1, Elevenmile Creek at Fla. Hwy. S-186	11/10	23	22	49
R-1, " " " " " "	11/12	17	15	24
No.2 Mill Recovery Area Effluent	11/10	*	*	*
" " " " " " " "	11/12	*	*	*
No. 1 Total Mill Ditch	11/12	*	1 to 2	4.3
Perdido Bay at Lillian Bridge	12/8	0.19	0.11	0.19
Perdido Bay ½ mi. Below Manuel Point	12/8	0.15	0.10	0.21

* - Unidentified
ND - None detected

APPENDIX A

PROJECT PERSONNEL

FEDERAL WATER QUALITY ADMINISTRATION

Billy H. Adams	Sanitary Engineer
Hugh C. Vick	Engineering Technician
R. P. Lewless	Chemist
Thomas B. Bennett,Jr.	Chemist

FLORIDA DIVISION OF AIR AND WATER POLLUTION CONTROL

Willard Hanks	Sanitary Engineer
John Shailer	Engineering Technician

Personnel from the St. Regis Engineering Technical Division also materially contributed to the completion of this survey.

APPENDIX B

SAMPLING PROCEDURE AND ANALYTICAL METHODS

SAMPLING PROCEDURE

Sample collection at the St. Regis Mill began during the day shift on November 10 and continued for seventy-two hours. For all practical purposes, the mill's sewer system is divided into two sewer systems -- one serving the original mill and the other serving later additions. Approximately 22 sampling locations are routinely sampled by mill personnel. Because of the work load and available BOD incubator space, however, sampling was limited to 10 of the established locations.

Sample collection within the mill was coordinated with work-shift sampling conducted by mill personnel. Except for grab samples collected at eight-hour intervals at sampling stations R-1, R-3 and R-9, all samples were collected at eight-hour intervals from continuous samplers.

The 5-day biochemical oxygen demand was the only analysis made by FWQA field personnel. Except for the BOD_5 analyses on 24-hour composite samples from sampling stations R-6 and R-10, all BOD_5 determinations were made on eight-hour composite samples.

All samples collected for total organic carbon analyses were either composite or grab samples collected at eight-hour intervals. Samples were transported to the Southeast Water Laboratory for TOC analyses.

Total suspended solids determinations were made by personnel assigned to the Florida Department of Air and Water Pollution Control Laboratory at Pensacola. All samples collected for suspended solids analyses were combined for a 24-hour composite sample.

Special foam samples for acids analyses were collected from sampling locations within the mill, and from Perdido Bay.

ANALYTICAL METHODS

1. Biochemical Oxygen Demand (Standard Methods for the Examination of Water and Wastewater, 12th Edition, 1965, pp. 415-421.)

2. Total Organic Carbon (FWPCA Methods for Chemical Analysis of Water and Wastes, November 1969).

A microsample of water is injected into a catalytic combustion tube heated to 950°C. The carbonaceous material is oxidized to carbon dioxide. An oxygen stream carries the CO_2 to an infrared analyzer sensitized to provide a measure of CO_2. The amount of CO_2 present is directly proportional to the concentration of carbonaceous material in the injected sample. The instrument used for this analysis is the Beckman Carbonaceous Analyzer, Model No. 915, dual channel.

3. Total Suspended Matter (Nonfilterable Residue) (Standard Methods for the Examination of Water and Wastewater, 12th Edition (1965), pp. 424-425).

4. Resin Acids

The samples were acidified and extracted with chloroform. The sample extracts were dried and esterified using the diazomethane procedure of Schlenk and Gellerman.[1/] The esterified extracts were adjusted to a certain volume and analyzed using a gas chromatograph equipped with dual flame ionization detectors. The identity of the resin acids was substantiated using mass spectrometry.

[1/]Schlenk, H. and J. L. Gellerman, 1960. "Esterification of Fatty Acids with Diazomethane on a Small Scale." Anal. Chem., 32: 1412.